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INVESTOR IN PEOPLE

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

JKU

REC'D 16 AUG 2000

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THE PATENT OFFICE

25 SEP 1999

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27SEP99 E479457-6 D01539
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The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference

BKCD/NS/DBN.104a

2. Patent applica
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9922693.8

25 SEP 1999

3. Full name, address and postcode of the or of each applicant (*underline all surnames*)

Trikon Holdings Limited
Coed Rhedyn
Ringland Way
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NP6 2TA

Patents ADP number (*if you know it*)

If the applicant is a corporate body, give the country/state of its incorporation

United Kingdom

4. Title of the invention

Method and Apparatus for Forming a film on a Substrate.

5. Name of your agent (*if you have one*)

Wynne-Jones, Laine & James

"Address for service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)

22 Rodnev Road
Cheltenham
GL50 1JJ

Patents ADP number (*if you know it*)

1792001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (*if you know it*) the or each application number

Country

Priority application number
(*if you know it*)

Date of filing
(*day / month / year*)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (*Answer 'Yes' if:*

Yes

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Description

8

Claim(s)

0

Abstract

0

Drawing(s)

7 6+6 11

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Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature

Wynne-Jones, Iaine & James

Date

Wynne-Jones, Iaine & James

24.9.99

12. Name and daytime telephone number of person to contact in the United Kingdom

Mr. B. Dunlop

01242 515807

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Method and Apparatus for Forming a film on a Substrate

In our pending application No. 9914879.3, the contents of which are hereby incorporated by reference, we described a method for forming a film on a substrate comprising:

(a) positioning the substrate on the support in a chamber;

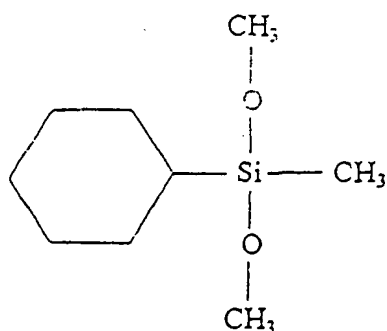
(b) supplying to the chamber in gaseous or vapour form a silicon containing organic compound and an oxidising agent in the presence of a plasma to deposit a film on the substrate; and

(c) setting (e.g. by annealing) the film such that carbon-containing groups are retained therein.

We suggest that the preferred oxidising agent is oxygen and indicate that the silicon-containing organic compound may be an alkylsilane or a tetralkylsilane.

Further experiments have now been carried out which suggest that methoxysilanes and in particular methoxymethylsilanes produce films with very low dielectric constants and may be particularly preferred.

Particularly good results have been achieved with cyclohexyldimethoxymethylsilane (CHDMMS) which has the following structure:



Experiments have also shown that a methoxysilane (e.g. CHDMMS may be able to be processed as in the above method described, but without any oxidising agent present in the plasma. It is supposed that this is because the Si-O bond already exists as part of the methoxy group.

Accordingly, according to another aspect the invention consists in a method of forming a film on a substrate comprising:

- (a) positioning the substrate on a support in a chamber;
- (b) supplying to the chamber in gaseous or vapour form an organic compound including an Si-O bond to deposit a film on the substrate; and
- (c) setting (e.g. annealing) the film such that carbon-containing groups are retained therein.

Preferably the compound is supplied in the presence of a plasma, but other energy sources may be utilised to cause appropriate deposition and these may be combined with spin-on techniques.

As before the platen or support temperature may be low, and initial experiments as shown in Figure 6 were carried out at various temperatures from 0°C to 70°C. Subsequent experiments have been carried out with a platen
5 temperature of 50°C rather than 0°C as previously described in British Patent Application No. 9914879.3.

The invention will be described with reference to the accompanying drawings, in which:

Figure 4 is a Fourier Transform Infra-Red (FTIR)
10 spectrum for a first process run without oxygen; and

Figure 5 is the equivalent FTIR for the process run with oxygen;

Figure 6 is a table showing initial experimental results using standard delivery systems for CHDMMS;

15 Figure 7 is a table showing experimental results using a syringe pump to deliver CHDMMS; and

Figures 8 to 10 are FTIR spectrum relating to certain experiments identified in Figure 7.

20 An experiment has particularly been carried out using cyclohexyldrimethoxymethylsilane (CHDMMS). As is reported below this has shown significantly reduced dielectric constants. It is anticipated that benefits will be found from many methoxysilane compounds such as tetramethoxysilane.

The experiments were carried out in a chamber substantially as shown in Figure 1 of our co-pending application 9914879.3 with an electrode gap spacings of 40mm and 20mm and the uniformity ring shield used for non plasma based processes removed. The CHDMMS was fed into the chamber using a syringe delivery system described in our co-pending application (filed and entitled "Delivery of Liquid Precursors to Semiconductor Processing Reactors", which is incorporated herein by reference), on the same date as opposed to a traditional low vapour pressure mass flow controller. This was done due to the fact that, as described below, CHDMMS could not be reliably delivered by conventional means as it has a relatively high boiling point (approximately 200°C) compared to most of the other precursor materials investigated in application 9914879.3.

All processes were run with plasmas applied to the showerhead. All wafers were 'set' by annealing for typically 30 minutes at approximately 480°C.

The following parameter ranges have been investigated:

Pressure	-	500 mT to 1500 mT
Power (380 kHz)	-	50 W to 750 W
Platen temperature	-	0°C to 70°C
CHDMMS flows	-	0.5 g/min to 1.5 g/min
Oxygen flows	-	0 to 200 sccm
Nitrogen flows	-	0 to 400 sccm
Peroxide flows	-	0 to 0.75 g/min

10 It will be appreciated that the relative flow rates are particularly relevant to the process. In general higher rates lead to higher deposition rates and thus a broad range of flow rates can achieve similar results. Thus values outside the above ranges may be applicable.

15 Two particularly preferred process examples are given below: one of these is with oxygen and one is without oxygen.

Process 1 (no O₂)	
Pressure	900 mT
Power	500 W
Platen temperature	50°C
CHDMMS flow	0.85 g/min
Nitrogen flow	200 sccm

20

Process 2 (with O₂)	
Pressure	900 mT
Power	250 W
Platen temperature	50°C
CHDMMS flow	0.85 g/min
Oxygen flow	50 sccm
Nitrogen flow	150 sccm

The resultant films were annealed and the post anneal results were as follows:

Process 1 (no O₂)	
Deposition rate	17000Å/min
Uniformity (max/min)	± 4%
Refractive index	1.370
Dielectric constant	2.55

Process 2 (with O₂)	
Deposition rate	9500Å/min
Uniformity (max/min)	± 5%
Refractive index	1.340
Dielectric constant	2.25

As can be seen the dielectric constants in each case are desirably low, but the "with oxygen" process is significantly advantageous.

Figures 4 and 5 show the respective FTIR spectra. It will be seen that they are substantially similar. The feature between 2500 and 2000 in Figure 5 is believed to - result from atmospheric (background) CO₂.

In fact, initial experiments were carried out using a CHMMS source consisting of a PTFE pot within an evacuated aluminium vessel which was heated to 150°C. The pot was connected by gas line to a gas mass flow controller suitable for H₂O with a conversion factor of 1.000. The RF power was applied to the showerhead with a spacing from the wafer of 40mm. The RF was 380khz continuous mode. Results from these experiments are shown in Figure 6. The

numbers in the CHMMS column are the nominal gas flow as measured by the mass flow controller however stable flows could not be achieved and therefore these results are for near random quantities of CHMMS being delivered to the process chamber. At this point experimentation was halted until a superior delivery system for this precursor could be developed.

CHMMS has a boiling point of 201.2°C, and a density of 0.940 g/cc. As it was noted in these experiments that CHMMS deposits a low k insulator without the addition of an oxidising agent it is therefore possible that it could be delivered as a liquid to a semiconductor wafer without a chamber being required (e.g. by well known 'spin-on' techniques) and then reacted either thermally or by plasma to form a low k ($k < 3$) insulator layer. The apparatus used may in effect deposit a liquid by vaporising the liquid precursor, delivering it as a vapour and then condensing it onto the wafer at a temperature below the boiling point of the precursor at that pressure. It is not yet clear if the reactions take place to the precursor on the wafer or at some other place, depositing reaction products onto the wafer.

Having developed a more suitable liquid delivery system which utilises a syringe pump, further experiments

were carried out as shown in Figure 7. From these experiments preferred processes were developed as further described here. FTIR for runs 13, 14 and 16-23 respectively are illustrated in Figures 8 to 10.

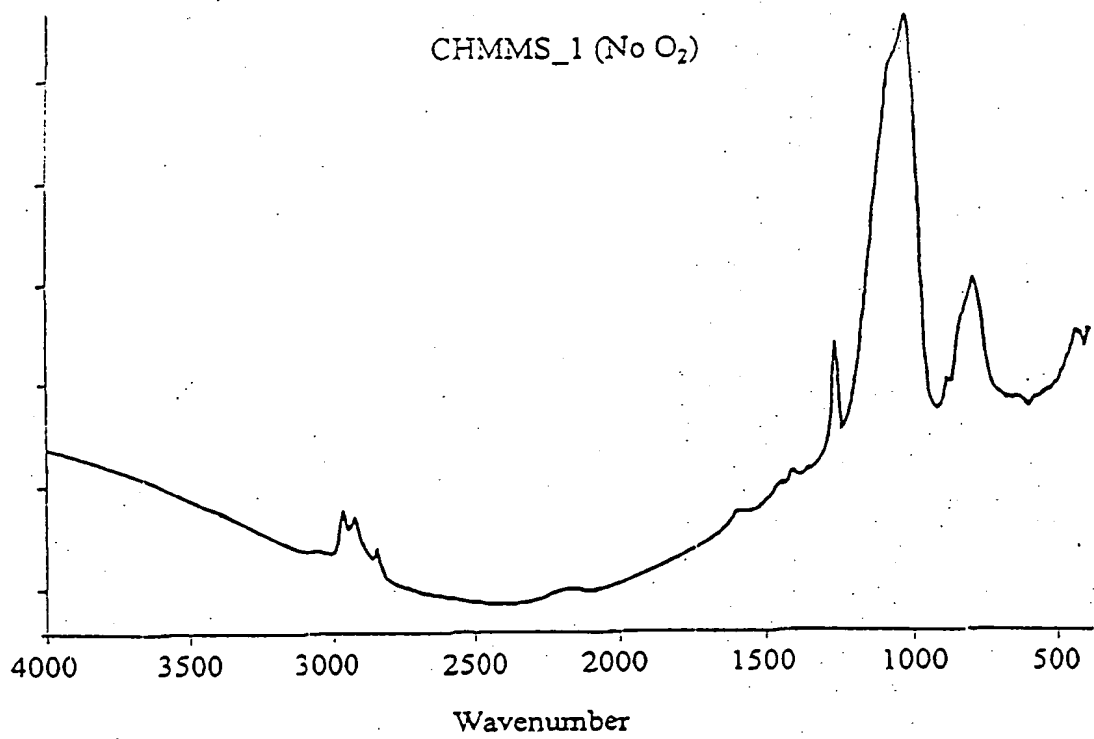


FIG 4

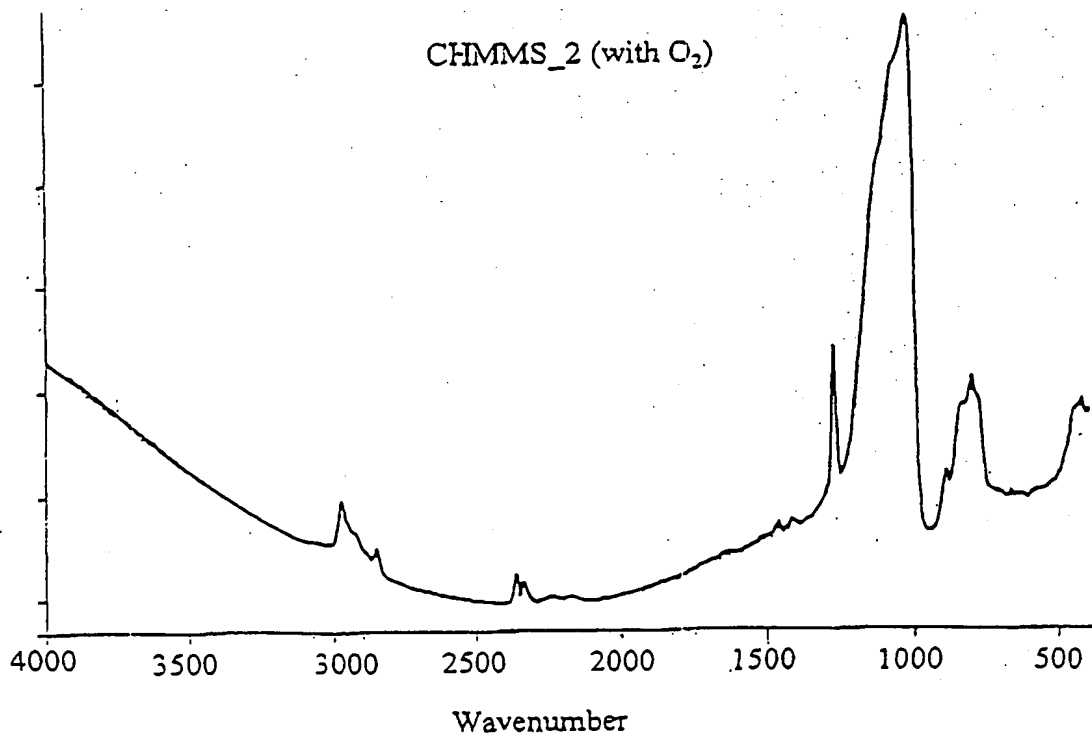


FIG 5

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D	Run No	CHMMS	H ₂ O ₂	O ₂	N ₂	Reactor (W)	Pressure (mT)	Platen (°C)	S/head (°C)	Dep Rate (Å/min)	Uniformity (%)	R _I	R.I Range	SiC/SiO	SiH/SiO	CH/SiO	K
Pot Refilled 24/05/99	1	1000	0.75	0	0	50	1500	70	100	1166 681	6.6 5.1	1.432 1.3702	0.0007 0.0074				
	2	1300	0.75	0	0	50	900	70	100	2542	6.5	1.3321	0.01				
	3	1300	0.7	0	0	100	900	70	100	1553	6.1	1.3676	0.015				
	4	1300	0.7	0	0	100	900	70	100	1450	8.5	1.5498	0.0125				
	6	1300	0.7	0	0	100	900	70	100	3916 2004	3.1 8.3	1.4736 1.5587	0.0023 0.0075				
	7	1500	0.7	0	0	100	900	70	100	3965 2097	22.1 22.5	1.5007 1.3749	0.005 0.005				
	8	1300	0.7	0	0	100	900	0	100	1592 716	15.5 13.9	1.4871 1.374	0.005 0.005				
	9	1500	0.7	0	0	100	900	0	100								
	10	1300	0.7	0	0	200	900	5	100	1176 738	27.3 25.6	1.4851 1.4064	0.0106 0.025				
	11	1300	0.7	0	0	200	900	0	100	-2000				0.0343 0.0191	0 0	0.1062 0.0069	
25/05/99	12	800	0.5	0	0	100	900	70	100	1731	15.9	1.4618	0.0163				
	13	800	0.4	0	0	500	900	70	100	9938	35	1.458		0.0183	0.0065	0.1102	
	14	800	0.4	0	0	250	900	70	100	2166	16.2	1.4569	0.0156				
	15	800	0	0	0	500	900	70	100	-10000	-30	1.45		0.0316	0.021	0.1715	
	16	800	0	0	0	250	900	70	100	-6000	-45			0.0299	0.0365	0.2756	
	17	800	0	0	0	250	900	70	100					0.0322	0.0376	0.303	
	18	800	0	0	0	250	900	70	100	5200							2.55
	19	800	0	0	0	250	900	70	100	7200							2.5-2.7
	20	800	0	0	0	250	900	40	100	5338	23.4	1.4938		0.05	0.039	0.2353	
	21	800	0	0	0	250	900	40	100	4200							3.2
	22	800	0	0	0	250	900	20	100	3641	14.4	1.4913	0.015	0.0395	0.027	0.237	
	23	800	0	0	0	250	900	20	100	6500							2.87

FIG 6

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Flowfill chamber depositions using Cyclohexyldimethoxymethylsilane

P727 - Flowfill chamber (Flow_1), 40mm el ctrode gap - Syringe delivery system

Process parameters

Bulk Film Properties

FIG 7

1	0.65	0	0	900	250	100	50	7224	10.6	1.4619		0.0357	0.0111	0.1347	Act. 1000mT, slight s/h
2	0.65	0	0	900	250	100	50	-	-	-		0.0345	0.0296	0.03672	Peeled off in FTS
3	0.65	0	100(1+8)	900	250	100	50	8190	6.5	1.4875		0.039	0.015	0.1545	Plasma dark red
4	0.65	0	50(1+8)	900	250	100	50	5810	22.8	1.4652		0.0354	0.0269	0.3499	Purple Plasma
5	0.65	0	50(1+8)	900	250	100	50	8289	13.1	1.51		0.0384	0.0107	0.131	
6	0.65	0	50(1+8)	900	250	100	50	8906	4.47	1.5089		0.0379	0.0139	0.1238	
7	0.65	0	100(1+8)	900	250	100	50	8717	2.37	1.4475		0.0364	0.0172	0.1886	
8	0.65	0	100(1+8)	900	100	100	50	2789	9.2	1.4775		0.0349	0.0367	0.3518	RI wafer
9	0.65	0	100(1+8)	900	500	100	50	12748	3.4	1.489					
10	0.85	0	100(1+8)	900	500	100	50	14222	1.77	1.538					
11	0.85	0	200(1)	900	500	100	50	14192	1.5	1.5228					
12	0.85	0	200(8)	900	500	100	50	14282	1	1.5444					
13	0.85	0	100(1+8)	500	500	100	50	9790	3.7	1.4895					
14	0.85	0	100(1+8)	500	500	100	50	11382	6.2	1.4468					RI wafer
15	0.85	0	200(8)	900	500	100	50	19116	5.6	1.4634					RI wafer
16	0.65	0	100(1+8)	900	250	100	50	10242	6.6	1.4558					
17	0.65	0	100(1+8)	900	250	100	50				3*				
18	0.85	0	200(8)	900	500	100	50				2.78*				K=2.4 post oven anneal
19	0.85	0	100(1+8)	500	500	100	50				2.82*				* Left overnight before measurement
20	0.65	0	100(1+8)	900	500	100	50				3.01*				K = 2.55 post oven anneal
21	0.85	0	100(1+8)	900	500	100	50	7869	7.8	1.5144					Grainy film, 5min FTS
22	0.85	0	100(1+8)	900	500	100	50	15697	5.7	1.5387					5min FTS
23	0.85	0	100(1+8)	900	500	100	50	14751	3.5	1.4737					5min FTS
24	0.85	0	100(1+8)	900	500	100	50	14345	0.9	1.4737					10min FTS
25	0.85	0	100(1+8)	900	500	100	50	14079	1.6	1.4582					30min FTS
26	0.85	0	100(1+8)	900	500	100	50	18864	4.5	1.4332					5min FTS
27	0.85	0	200(8)	900	750	100	50	17841	7.2	1.4327					5min FTS
28	0.85	0	200(8)	900	250	100	50	11511	6.3	1.4263					5min FTS
29	0.85	0	200(8)	900	500	100	50	15565	3.5	1.4856		0.0317	0.0193	0.1366	5min FTS
30	0.85	0	200(8)	900	500	100	50	14807	3.1	1.4575		0.0336	0.0096	0.0785	30min FTS
31	0.85	0	200(8)	900	750	100	50	16898	3.8	1.503		0.0284	0.016	0.1418	5min FTS
32	0.85	0	200(8)	900	250	100	50	11658	11.5	1.499		0.0342	0.0338	0.3437	5min FTS
33	0.85	0	200(8)	900	500	100	50				2.56				Depped with 30min FTS + Cap
34	0.85	0	200(8)	900	500	100	50				2.66				Depped with 30min FTS
35	0.85	0	200(8)	900	500	100	50	17106	3.7	1.4552		0.0309	0.0199	0.1562	5min FTS
36	0.85	0	200(8)	900	500	100	50	17194	3.7	1.458		0.031	0.0202	0.1498	5min FTS
37	0.85	0	200(8)	1200	500	100	50	24708	2.2	1.5316		0.0311	0.0196	0.1338	5min FTS
38	0.85	0	200(8)	600	500	100	50	9933	1	1.5109		0.0353	0.018	0.1283	5min FTS
39	0.85	0	200(8)	600	250	100	50	7128	0.87	1.5296		0.035	0.0248	0.258	5min FTS
40	0.85	0	150(8)	900	250	100	50	9652	3.8	1.4575		0.0285	0.0162	0.2006	5min FTS
41	0.85	0	150(8)	900	500	100	50	18448	3.8	1.4209		0.0203	0.009	0.0839	Feint powder showerhead pattern
42	0.85	0	250(8)	900	500	100	50	~1.8µm	-	-		0.0209	0.0058	0.0572	Entire film cloudy
43	0.85	0	0	900	500	100	50								

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94	0.85	50	0	900	500	100	50	21408	4.6	1.4199	2.49	0.0273	0.0076	0.0725	30min FTS, cap
95	0.85	50	150(8)	900	250	100	50								30min FTS, cap
96	0.85	50	150(8)	900	250	100	50				2.48				30min FTS, cap
97	0.85	25	175(8)	900	250	100	50	9917	8.8	1.4521		0.0342	0.0093	0.1091	5min FTS
98	0.85	25	175(8)	900	250	100	50	9848	11.7	1.4592		0.0334	0.0117	0.1441	5min FTS
99	0.85	25	175(8)	900	250	100	50				2.437				5min FTS, cap
100	0.85	25	175(8)	900	250	100	50				2.286				60min FTS, cap
101	0.85	25	175(8)	900	250	100	50				2.426				30min FTS, cap
102	0.43	0	100(8)	900	500	100	50	12080	7.6	1.5107		0.0239	0.0093	0.096	5min FTS Showerhead dots
103	0.43	0	100(8)	900	500	100	50	12502	5.1	1.5081		0.025	0.0119	0.0116	5min FTS Showerhead dots
104	0.64	0	150(8)	900	500	100	50	20470			2.9				30min FTS cap
105	0.85	0	400(8)	900	750	100	50	14074	3.5	1.4983		0.0282	0.0149	0.0912	5min FTS
106	0.85	0	400(8)	900	750	100	50	13930	2.4	1.496		0.0278	0.0143	0.0771	30min FTS
	0.85	0	400(8)	900	750	100	50				2.72				30min FTS CAP

CHAMBER SPACING CHANGED TO 20MM

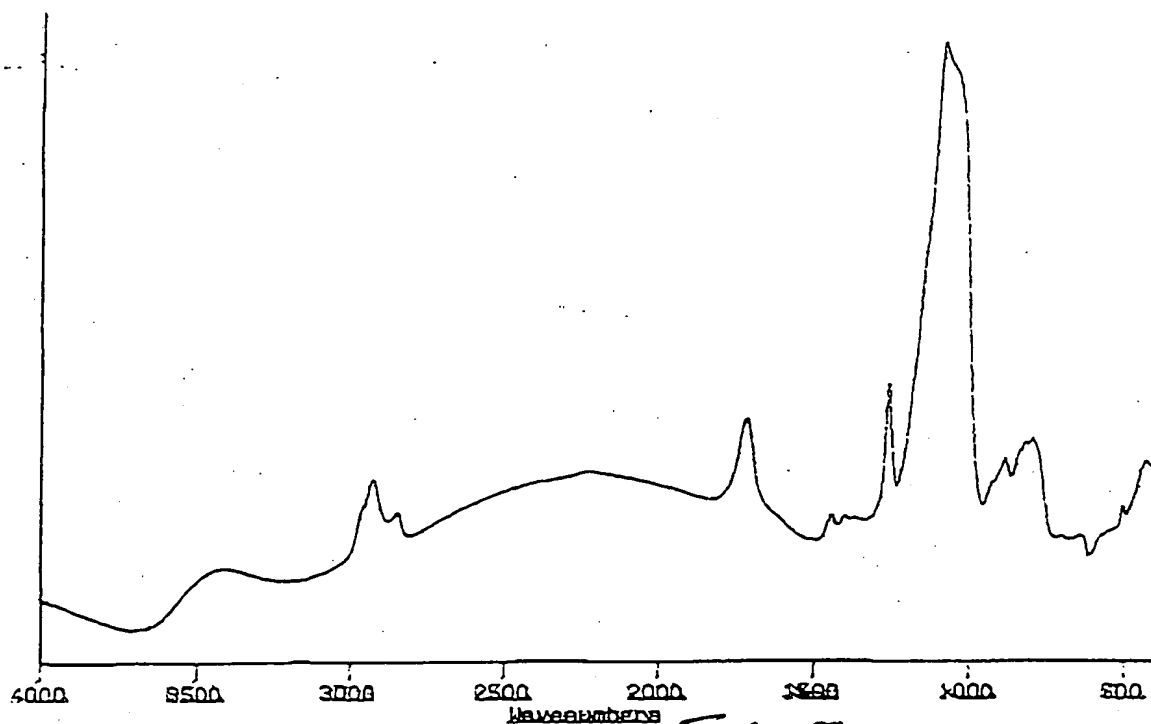
108	0.85	0	200(8)	900	500	100	50	17626	-	1.3437					30min FTS
109	0.85	0	200(8)	900	500	100	50	21765	8.4	1.3654					30min FTS, RI Water
110	0.85	25	175(8)	900	250	100	50	11436	17.6	1.3713					30min FTS
111	0.85	25	175(8)	900	250	100	50	12828	13.6	1.3888					30min FTS, RI Water
112	0.85	0	200(8)	600	500	100	50	14280	11.1	1.447					30min FTS, RI Water
113	0.85	0	200(8)	600	500	100	50	12185	3.4	1.3756					30min FTS
114	0.85	0	200(8)	400	500	100	50	9049	2.8	1.4745					30min FTS
115	0.85	0	200(8)	400	500	100	50	10620	8.6	1.4549					30min FTS, RI Water
116	0.85	0	200(8)	400	500	100	50	9073	3.5	1.4524		0.0255	0.0143	0.0724	30min FTS
117	0.85	0	200(8)	900	500	100	50	14852	2.3	1.4384		0.0343	0.0096	0.0865	30min FTS
118	0.85	0	200(8)	900	500	100	50				2.556				30min FTS, Cap
119	0.85	0	200(8)	400	500	100	50				2.76				30min FTS, Cap
120	0.85	0	200(8)	900	500	100	50	11633		1.4334		0.0363	0.0094	0.0751	30min FTS, Stress = 8.78E8T
121	0.85	0	200(8)	900	250	100	50	8613		1.399		0.0386	0.0091	0.01019	30min FTS, Stress = 7.31E8T
122	0.85	25	175(8)	900	250	100	50	9207		1.3954		0.0329	0.0069	0.0493	30min FTS, Stress = 9.856E8T
123	0.85	50	150(8)	900	250	100	50	10515		1.381		0.0313	0.007	0.0502	30min FTS, Stress = 9.325E8T
	0.85	15	185(8)	900	250	100	50	10640		1.3807		0.034	0.0051	0.0556	30min FTS, Stress = 9.041E8T
	0.85	75	125(8)	900	250	100	50	11727		1.3597		0.0295	0.0042	0.0344	30min FTS, Stress = 8.544E8T
126	0.85	100	100(8)	900	250	100	50	12598		1.3481		0.0293	0.0056	0.0336	30min FTS, Stress = 8.778E8T
127	0.85	25	175(8)	900	250	100	50	9206		1.3718		0.0331	0.0061	0.0466	30min FTS, Stress = 8.025E8T
128	0.85	15	185(8)	900	250	100	50				2.414				30min FTS
129	0.85	75	125(8)	900	250	100	50				2.4				30min FTS
130	0.85	100	100(8)	900	250	100	50				2.49				30min FTS
131	0.85	25	175(8)	900	250	100	50				2.41				30min FTS
132	0.85	35	165(8)	900	250	100	50				2.48				30min FTS
133	0.85	15	185(8)	900	250	100	50				2.43				30min FTS
134	0.85	30	170(8)	900	250	100	50				2.45				30min FTS

FIG 7 (cont'd)

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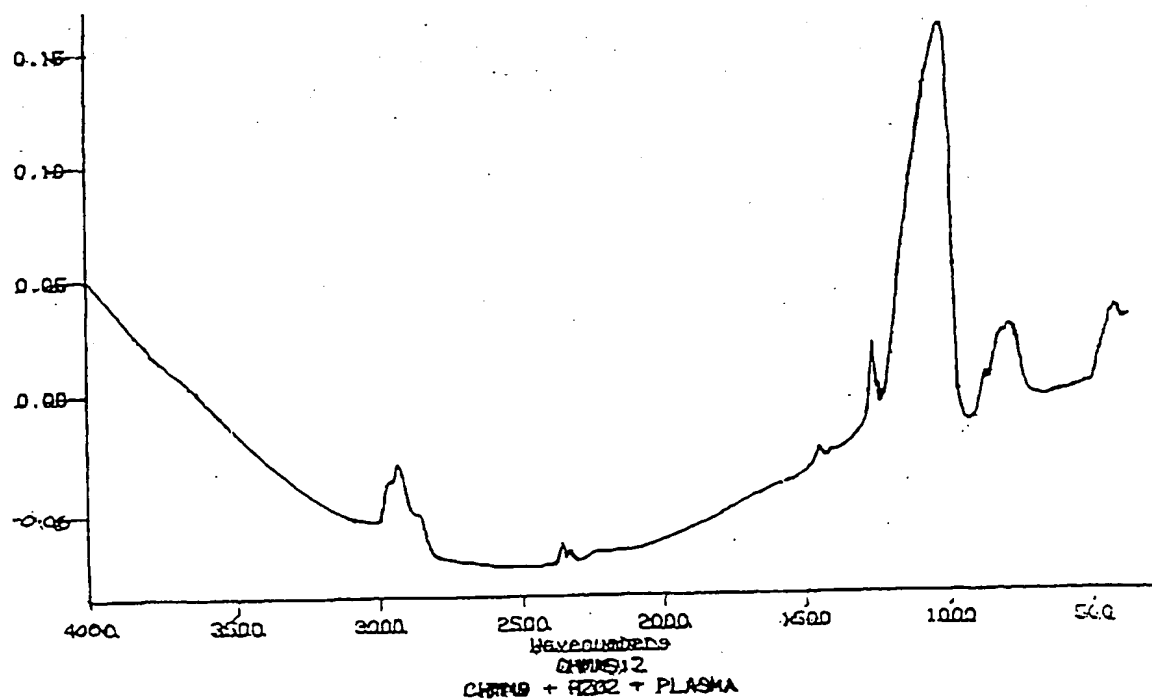
FTIR Spectra

1.) 800CHMMS, 0.4g/min H₂O₂, 900mT, 250W as deposited



2.) 800Sccm CHMMS, 0.4g/min H₂O₂, 900mT, 500W

FIG 9



CHMMS + H₂O₂ + PLASMA

FIG 8

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3.) 800Sccm CHMMS, 900mT, 250W

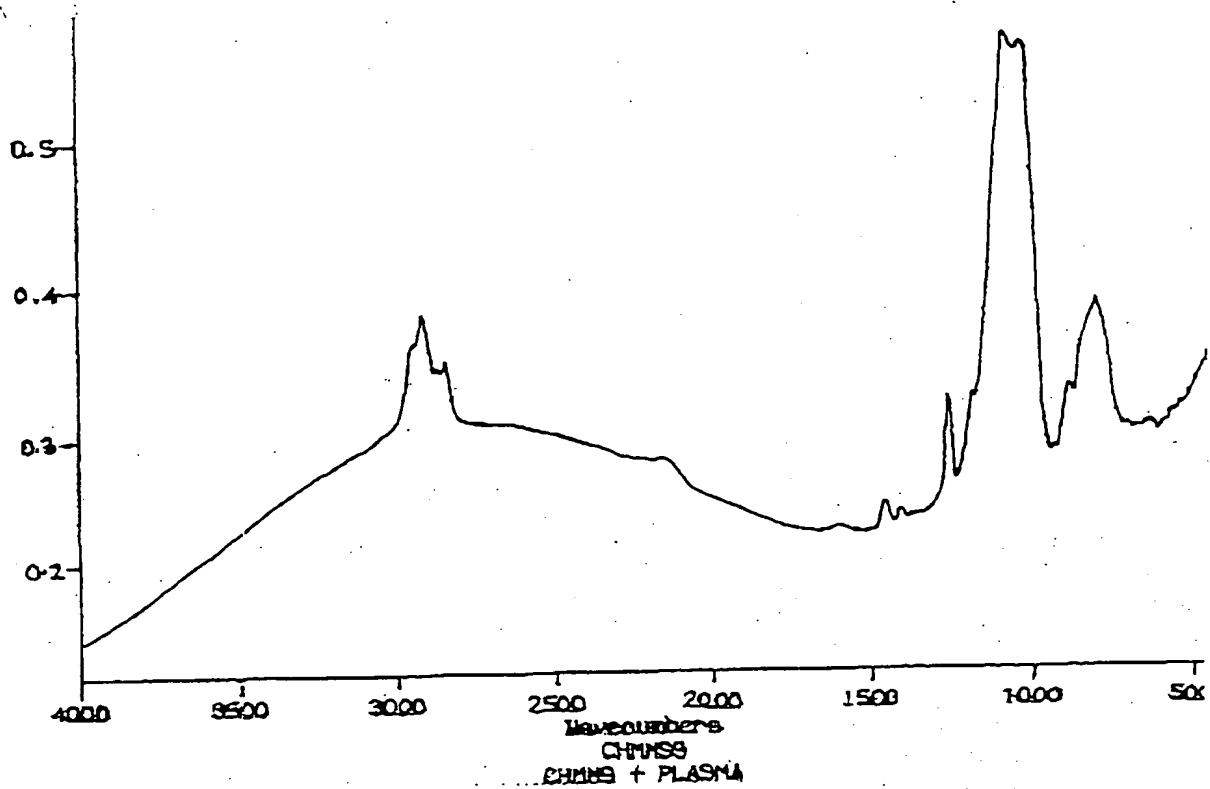


FIG 10

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